Investigations of the cleanability of polymer filter media

C. Leipert, H. Nirschl*

Filter media cleaning plays a central role in filtration processes. Efficient cleaning of these filter media prevents cross-contamination and increases the product quality. This article therefore deals with the cleaning behaviour of filter fabrics. The focus is above all on the physical fundamentals in cleaning. The performed examinations are supposed to provide information about the cleanability of filter fabrics with different geometries (mesh width and thread diameter). The influence of various materials (PP, PET, PA6) and different particle sizes on the cleanability of the filter cloth will also be discussed.

1. Introduction

Numerous examinations have been performed on the cleanability of pipelines. Graßhoff /1/ dealt with the cleaning behaviour of cylindrical dead spaces. He investigated different flow patterns in rectangular branched pipeline systems. He concluded that the dead space problem would have to possibly be solved by installing flow dividers that guide a subflow directly into the dead space /1/.

Lelieveld /2/ discussed process examples of frequently occurring CCPs (Critical Control Points). Above all, he took a closer look at static and dynamic seals. Demands on seals in aseptic fields are especially high. They are supposed to prevent the penetration of microorganisms from the outside as well as leakage of product from the process chamber. Lelieveld detected that the O-ring between stainless steel flanges expands to such an extent with rising temperature that it protrudes over the outside edge of the flanges. After cooling down, the shape of the sealing ring changed and allowed penetration of microorganisms into the product chamber. Changes in pressure resulted in a shift of the O-ring towards the side that comes into contact with the product. Microorganisms are then also transported into the process chamber. Individual static seals are very effective to block off leaks, but...
cannot prevent the penetration of bacteria. Benzécri /3/ investigated the cleanability of an eccentric screw pump that is frequently used in the food industry. His goal was to develop an exact method to test the cleanability of process facility components. For contamination he used spores of the B. cereus stem. The test method provided good and exact results. However, it can only be applied to a limited extent for components in the food sector. After all, using nutrient agar to reach hard to access places in components permanently in contact with foodstuffs is very critical. Hofmann /4/ considered the cleaning process as mass transfer and its adaptation as qualification method for the cleanability of production facilities.

Contrary to this, Bobe /5/ investigated particle removal on technically relevant surfaces. One part of his work focused on examining the detachment behaviour of particles on surfaces with different properties during flowing. He examined the influence of surface roughness, the surface energy, and investigated different surfaces with exactly defined structures. These structures were produced by means of lithographic etching. One roughness structure consisted of grooves with different widths vertical to the flow direction. However, another roughness structure investigated by Bobe showed column-shaped roughness of varying width. He examined two particle sizes (1.16μm and 5.71μm). In each produced structure, the "small" particles were located between the elevations, the larger ones on the other hand only fitted into one single structure between the elevations. Bobe found out that the structure of a surface and the size relation of groove width structure to the particle size determines cleanability much more than a measured roughness parameter. On the other hand, he ascertained that surfaces with lesser surface energies are better to clean.

Weigl /6/ investigated the adhesiveness of polystyrene particles, yeasts and glass beads on surfaces with different degrees of roughness. Amongst other things, he found out that more force must be applied when separating particles from a rough surface than from a smoothly polished surface. The contact surfaces increases in this case, and thus the adhesiveness of the particles. Therefore, the demand for even smoother surfaces does not seem to be expedient.

Stahl /7/ was the first one to deal with the cleaning of filter media. He used a microbiological test method by the EHEDG (European Hygienic Design Group) to assess the cleanability of filter media. As we can discern from the literature, remarkable advancements have been made in the hygiene sector for pipelines, pumps and seals in the past years. The EHEDG has developed many guidelines and instructions concerning the hygiene-conform construction of these parts. However, there are no guidelines whatsoever that have to be observed for optimum cleanliness in the field of filter media cleaning. Large quantities of acidi and alkaline cleaning agents are often used to achieve the desired degree of cleanliness. Particles still remain on the filter medium after cake removal or discharge in filtration processes. This is why this article deals with the investigation of physical principles in filter medium cleaning.

### 2. Theory

#### 2.1 Adhesion force

The adhesion force between surface and contamination has to be overcome in order to make hydrodynamic cleaning successful. Soluble substances as well as particles may be regarded as contamination.

The physics of adhesion will be explained in more detail in the following. Schubert /8/ classified the adhesion forces according to binding mechanisms with and without material bridges. According to him, bonding mechanisms with material bridges are sintering processes, hardening of crystalline dissolved substances in the contact area, fluid bridges and capillary fluid. Without material bridges, van der Waals forces, electrostatic forces and positive-fit bondings are responsible for adhesion.

As the cleaning of the filter media takes place in watery media in the scope of this examination, forces through watery liquid bridges are omitted. Adhesive forces through sintering, crystallisation or positive fit were also not generated. According to this, only the van der Waals and electrostatic forces play a role in these examinations. Electrostatic forces occur with charged particles and walls, while van der Waals forces occur every time. Therefore, adhesion in watery environments can only be effected by electrostatic and van der Waals forces /9/.

#### 2.2 Interaction of electrostatic and van der Waals forces

As mentioned above, adhesion is comprised of van der Waals and electrostatic forces.

$$ F_{\text{H}} = F_{\text{vdW}} + F_{\text{el}} $$

Here, \( F_{\text{H}} \) is the adhesive force, \( F_{\text{vdW}} \) the van der Waals force and \( F_{\text{el}} \) the electrostatic force.

The van der Waals attraction can be explained with quantum-mechanical processes. The electron movement in an atom or molecule leads to charge fluctuations that result in a temporally changeable dipole. With a sufficiently small distance, the formation of this fluctuating dipole results in the attraction of two adjacent particles. The extent of the van der Waals force depends on the particle size, the materials of the bonding partners, the intermediate medium (in this case water) and the distance between the bonding particles /8; 10/.

Electrostatic force occurs between charged particles and charged surfaces. The charge of the surface may either lead to attraction (surfaces with different charges) or repulsion (surfaces with the same charge). The electrostatic force can be calculated as follows using the sphere-disk model:

$$ F_{\text{el}} = \pi \cdot d \cdot \delta \cdot k \cdot T \cdot \rho_{\text{a}} \cdot \delta \cdot \frac{B_{\text{el}}}{a} \cdot \frac{\tan \left( \frac{z_{1} \cdot \varepsilon_{1}}{4 \cdot k \cdot T} \right) - \tan \left( \frac{z_{2} \cdot \varepsilon_{2}}{4 \cdot k \cdot T} \right)}{\tan \left( \frac{\varepsilon_{1}}{4 \cdot k \cdot T} \right)} $$

Here, \( A \) is the contact surface, \( T \) the absolute temperature, \( \rho_{\text{a}} \) the ambient density, \( z \) the ionic valence, \( \zeta \) the zeta potential of the respective substance, \( \delta \) the thickness of the diffusive layer, \( a_{\text{H}} \) the adhesion distance, and \( a + \delta \) the distance between the two bonding partners. If both surfaces repulse each other (particles and wall have the same charge), the bonding partners can usually not approach each other to such an extent that the van der Waals force comes into effect. If the bonding partners attract each other, van der Waals forces occur additionally /9/.

#### 3. Material and methods

##### 3.1 Filter media

The investigated materials are shown in table 1, table 2 and table 3. These concern fabrics made of polypropylene (PP), polyamide 6 (PA 6) and polyethylene terephalate (PET) in linen weave. One aim was to describe the influence of the filter geometry on the cleanability of the filter medium in greater detail. For this purpose, filter media with constant mesh widths but different thread diameters and filter media with constant thread diameter and varying mesh widths were investigated (see tables 1 and 2). The selected filter media in table 3 are supposed to provide information about the influence of the material.

The selection criteria for the filter media mainly referred to the examination of the geometry parameters.

##### 3.2 Test performance

3.2.1 Contamination of the filter media

The filter media were contaminated with fluorescence-marked particles by Microparticles. These were monodispersed melamine resin particles with a very narrow particle size distribution.
Particles with the sizes 5 and 10 μm were used.

After the discharge of the filter cake, a lot of particles still remain on the filter medium in filtration processes. This status is to be assumed in these examinations. The particle charge prior to the cleaning test was between 800 and 1,000 particles on a surface of 24 mm². The fabric was only charged with a suspension here, a cake did not form. Only individual particles deposited on the filter medium. The number of particles located on the surface of the filter medium before and after the cleaning process was determined with a fluorescence microscope and direct counting. The particle residue corresponds to the ratio between the particle count on the filter after and before cleaning.

3.2.2 Perfusion of the filter media

The contaminated filter media were perfused at three different flow speeds (0.6 m/s, 0.8 m/s and 1 m/s). The rinsing time was constant in all tests (20 s). Demineralised water was used as cleaning agent in the examination of the geometry, particle size and material. Weak acids and/or bases were used for the test investigating the influence of the zeta potential.

The fluorescence microscope images of the fabrics after perfusion provided information about the remaining particles on the fabric surface. The particle retention was thus determined using the following formula:

\[ R = \frac{N_{\text{after}}}{N_{\text{vor}}} \times 100\% \]  

Whereby \( N_{\text{after}} \) stands for the particle count after perfusion and \( N_{\text{vor}} \) for the particle count before perfusion.
Fig. 1 shows the schematic structure of the through streaming plant.

The system consists of a pressure tank (1) (volume: 99 litres) and a filter retainer (2). The cleaning agent is transported from the container (1) through the filter retainer onto a scale (3). Two pressure sensors are additionally attached to the filter retainer in order to measure the pressure loss during perfusion of the filter medium. The flow velocity through the filter medium can be adjusted via the tank pressure.

4. Results and discussion
4.1 Dependence of cleaning on the geometry of the filter medium

The following fig. 2 shows the particle residue on the filter medium in dependence on the perfusion speed. The residue on the filter medium declines with rising flow velocity, because higher flow velocities lead to higher wall shear stress on the filter thread and on the particles. The particles are therefore exposed to higher forces with increasing flow flow velocities.

The applied filter media had the same thread diameter d but different mesh widths w (see fig. 2). The cleaning result of the filter deteriorates with increasing mesh width (residue R increases). This becomes especially apparent in filter media with the mesh widths w = 590 μm and w = 1180 μm. The difference in particle retention on the filter is almost twofold.

The filter medium with the smaller mesh width has a smaller free surface than the filter medium with the larger mesh width. The washing fluid must be accelerated faster with the smaller mesh widths in a constant mass flow than with the larger ones. Accordingly, larger forces impact on the particles in the smaller mesh widths, which lead to easier detachment of the particles.

4.2 Dependence of cleaning on the particle size

Fig. 4 shows the influence of the particle size on cleaning. It is evident that the degree of cleaning increases the larger the particles are. Enlarging the particles thus has a positive effect on the cleaning behaviour. The reason is that the adhesion forces (van der Waals forces, electrostatic forces) only rise proportionally to the particle diameter, while the separating forces (meaning: buoyancy forces, resistance forces and the moment of surface tension acting on the impact surface of the particle) rise proportionally to the squared particle diameter as the model by Burdick /11/ shows. Coarser contamination can therefore be separated easier than finer contamination.

4.3 Dependence of cleaning on the filter fabric

The filter material is another factor that greatly influences cleaning. Three materi-

Fig. 2: Influence of the mesh width on cleanability (filter fabric acc. to table 2)
Fig. 3: Influence of the thread diameter on cleanability (filter fabric acc. to table 1)
Fig. 4: Influence of the particle size on cleanability (filter fabric consisting of PP, w = 297 μm, d = 215 μm)
als were available for these tests: PP, PET and PA 6. As fig. 5 shows, the materials PP, PET and PA 6 do not have a significant influence on cleanability. The retention with all three materials is 25 to 30 % in the scope of measuring accuracy.

These results can be confirmed by examining the adhesion forces of these three materials. This was determined with an ultracentrifuge. In this test, particles located on single threads of the three materials were installed in a special retainer in the ultra-centrifuge and stressed at rising rpm. The filter threads were evaluated after each agitation speed under a fluorescent microscope. The separation forces were calculated via the centrifugal force. The separation forces were as follows: PET 95.3 nN, PA 6 95.8 nN and PP 57.9 nN. The results show that the separation forces of the system particle/PA 6 and particle/PET were very close in the scope of measuring accuracy as the results in fig. 5 lead to expect. However, the system polypropylene/particle showed a much lower separation force. Accordingly, it must therefore be easiest to separate the particles from polypropylene tissue. However, this is not the case in fig. 5. Polypropylene has almost the same cleaning result as PA6 and PET. The reason for this might lie in the measuring inaccuracy of the cleaning tests.

4.4 Dependence of cleaning on the zeta potential

Fig. 6 shows cleaning tests with 5 μm particles for a filter medium consisting of PA 6. These tests were performed in an ultrasound bath at different pH values. The pH value solutions were produced with acetic acid resp. caustic soda without adding any electrolytes. Fig. 6 shows that PA 6 achieves the best cleaning result in the alkaline range.

To clear up this circumstance, zeta potential measurements were conducted in dependence on the pH value on the filter media as
well as the particles. The zeta potential measurements were performed with a flow potential measuring apparatus at the Technical University of Kaiserslautern. The results are displayed in fig. 7. The isoelectric point of the particles is located at a pH value of 6.1 while that of the filter medium has a pH value of 4.2. The figures show that the zeta potential is positive in the acidoic range and negative in the alkaline range. This means that the material surface is negatively charged in the alkaline range (OH ion excess) and positive in the acidoic range (H3O+ ion excess). The line range (pH value of 6.1) and positive in the alkaline range. This means that the material surface is negatively charged in the alkaline range (OH ion excess) and positive in the acidoic range (H3O+ ion excess). The zeta potentials of the particles and the filter surface have different prefixes between the two isoelectric points (meaning between pH 4.2 and 6.1). In this case, the particle surface is positively charged while the filter surface carries a negative charge. Due to the fact that opposite charges attract, the adhesiveness is boosted by electrostatic attraction. The energy barrier that has to be overcome in order to separate the particles increases accordingly. Fig. 7 also confirms this fact. The largest particle residue on the filter surface is encountered in the range between pH 4.2 and 6.1.

With pH values lower than 4.1, the zeta potentials of the filter surface and the particle surface are negative. Both surfaces have the same type of charge. Once again, electrostatic repulsion takes place, which in turn leads to a reduced energy barrier that has to be overcome. In these two boundary areas, there is less retention than between pH 4.2 and 6.1 as shown in fig. 6.

Knowledge of the zeta potential of contamination and filter surface would make it possible in the future to make statements concerning the required pH value of a cleaning agent to be used.

5. Summary and outlook

As the examinations show, the three filter media materials (PP, PET, PA 6) do not show any difference in cleanliness. This result is evident, as all three materials are polymers and thus have similar substance properties and/or boundary surface properties. Therefore, only the pH and temperature resistance should play a role in the selection of the filter material. Likewise, much stronger forces have to be applied with small particle sizes to remove these from the filter surface.

As also shown, filter media with smaller mesh are much easier to clean than filter media with large mesh widths, while filter media with larger thread diameters can be cleaned better than those with smaller diameters. This is due to the larger lateral surface area of the filter medium. The larger the lateral surface area with constant filtration surface, the higher the required acceleration of the cleaning fluid in the pores.

However, the highest cleaning rate ever achieved in tests with demineralised water was merely approx. 88%. As the tests with pH variations in the cleaning fluid have shown, the cleaning rate may increase up to 98.5% when selecting the right pH value.

The cleaning of colloidal particles may be problematic. Because the smaller the particles, the higher is the adhesion force, which makes it more difficult to detach the particles from the filter surface. Mere backstreaming will not be sufficient here. This is why it will be necessary to boost filter medium cleaning with pulsed flow.

6. Acknowledgements

The authors would like to thank the AiF for financing the project, the Sefar company for providing the filter media, and the Professor for Mechanic Process Engineering at the Technical University Kaiserslautern, Professor Dr. Ing. Siegfried Ripperger, for performing the flow potential measurements.

Literature

/2/ Lelieveld, H. L. M., M.A.C.P and hygienic design, Food Control, 5 (1994), no. 3, p. 140-144
/4/ Hofman, J.: Stoffübergang bei der Reinigung als Qualifizierungsverfahren der Reinigbarkeit, Dissertation (2007), Technical University Munich
/5/ Bobe, U.: Die Reinigbarkeit technischer Oberflächen im immergrierten System dissertation (2008), Technical University Munich
/6/ Weigl, B.: Trennkräfte zwischen Mikroorganismen, Partikeln und Oberflächen, dissertation (2003), Technical University Munich
/8/ Schubert, H.: Grundlagen des Agglomerierens, Chemie Ingenieur Technik, 51 (1979), no. 4, p. 266-277
/A/ Nassauer, J.: Adsorption and Haftung an Oberflächen and Membranen, Technical University Munich (1985)